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- Fourier transform ion cyclotron resonance (FTICR) mass spectrometry was used to obtain information about the reactivity and physical properties of gaseous cluster ions, and to develop various mass spectrometric surface and bulk analysis techniques. New values for electron affinities and ionization potentials of many different semiconductor clusters were determined. Ultra-high resolution glow discharge FTICR mass spectrometry was developed to better characterize solid materials.
14. SUBJECT TERMS Electrospray Ionization, Organometallic complexes, Fourier transform ion cyclotron resonance mass spectrometry, Clusters, Ionization Potentials, Electron Affinities, Glow Discharge, Ultra-high Mass Resolution 15. NUMBER OF PAGES 11
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FINAL REPORT

Mass Spectrometric Studies of Clusters and Surfaces

by

Prof. John R. Eyler
Principal Investigator

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August 29, 1994

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Objective

The objective of the research carried out under this contract was to obtain information about the physical properties and reactivity of gaseous cluster ions, and to develop various mass spectrometric surface and bulk analysis techniques (such as laser desorption - LD - and glow discharge - GD). Better understanding of cluster ion properties and reactivity makes possible further development of this important form of matter for catalysis and synthesis of new materials. Improved mass spectrometric analysis of surface and bulk materials can enhance the design and synthesis of solid state materials with new superconducting properties and lead to much higher capabilities of solid state devices.

Approach

The work carried out under this grant utilized the unique capabilities of Fourier transform ion cyclotron resonance (FTICR) mass spectrometry to trap ions and study their structures and reactivities, as well as to determine their masses.

Conclusions

The superior ability of FTICR to follow ion/molecule reactivity was used to determine rate coefficients and product distributions of metal and non-metal cluster ion reactions. Charge transfer reactions were used to determine the adiabatic ionization potentials (IP's) and electron affinities (EA's) of gas-phase clusters. New sources of metal cluster ions were explored, such as metal oxides and metal-containing solid materials. A two-laser infrared multiphoton dissociation technique was used to obtain infrared spectra of gaseous ions. Electrospray ion generation/injection was used to transfer multiply-charged metal clusters surrounded by a ligand shell from solution into the FTICR cell for determination of their reactivities and other properties. Glow discharge sources were coupled in two different ways to FTICR mass spectrometers to permit ultra-high mass resolution studies which allowed contaminants or isobaric interferences to be identified by separating their peaks in the mass spectrum from those of the sample of interest. The highest mass resolution ever attained in GD mass spectrometry was reported.

Publications/Patents/Presentations/Reports Under this Grant

Published Papers in Refereed Journals

D. M. Branan, N. W. Hoffman, E. A. McElroy, D. L. Ramage, M. J. Robbins, J. R. Eyler, C. H. Watson, P. deFur, and J. A. Leary, "A Comparison of Laser Desorption and Fast Atom Bombardment Mass Spectra of a Series of $\text{Rh}(\text{PPh}_3)_2(\text{CO})\text{Y}$ Complexes," *Inorg. Chem.* **1990**, 29, pp 1915-1919.

A. R. Katritzky, Z. Dega-Szafran, C. H. Watson, and J. R. Eyler, "Collisionally Activated Dissociation of 1-Benzoyloxypyridinium Cations", *J. Chem. Soc. Perkin Trans. II* **1990**, pp. 1051-1057.

J. A. Zimmerman, S. B. H. Bach, C. H. Watson, and J. R. Eyler, "Ion/Molecule Reactions of Arsenic and Phosphorus Cluster Ions: Ionization Potentials and Novel Reaction Pathways," *J. Phys. Chem.* **1991**, 95, pp. 98-104.

J. A. Zimmerman, C. H. Watson, and J. R. Eyler, "Multiphoton Ionization of Laser-Desorbed Neutral Molecules in a Fourier Transform Ion Cyclotron Resonance Mass Spectrometer," *Anal. Chem.* **1991**, 63, pp. 361-365.

* J. A. Zimmerman, J. R. Eyler, S. B. H. Bach, and S. W. McElvany, "'Magic Number' Carbon Clusters: Ionization Potentials and Selective Reactivity," *J. Chem. Phys.* **1991**, 94, pp. 3556-62.

C. H. Watson, J. A. Zimmerman, J. E. Bruce, and J. R. Eyler, "Resonance Enhanced Two-Laser Infrared Multiple Photon Dissociation of Gaseous Ions," *J. Phys. Chem.* **1991**, 95, pp. 6082-6.

D. K. Bohme, S. Wlodek, J. A. Zimmerman, and J. R. Eyler, "Formation of $\text{C}_{10}\text{H}_8^+$ from the Benzene Radical Cation: A Case for the Growth of Polycyclic Aromatic Hydrocarbon Ions by Ion-Molecule Reactions in the Gas Phase?," *Int. J. Mass Spectrom. Ion Proc.* **1991**, 109, pp. 31-47.

C. M. Barshick and J. R. Eyler, "A Glow Discharge Ion Source with Fourier Transform Ion Cyclotron Resonance Mass Spectrometric Detection," *J. Am. Soc. Mass Spectrom.* **1992**, 3, pp. 122-127.

M. A. Cheeseman and J. R. Eyler, "Ionization Potentials and Reactivity of Coinage Metal Clusters," *J. Phys. Chem.* **1992**, 96, pp. 1082-9.

J. E. Bruce and J. R. Eyler, "Probing Trapped Ion Energies via Ion-Molecule Reaction Kinetics: Fourier Transform Ion Cyclotron Resonance Mass Spectrometry," *J. Am. Soc. Mass Spectrom.* **1992**, 3, 727-33.

C. M. Barshick and J. R. Eyler, "An Improved Ion Guide for External Ion Injection in Glow Discharge - Fourier Transform Ion Cyclotron Resonance (GD-FTICR) Mass Spectrometry," *J. Am. Soc. Mass Spectrom.* **1993**, *4*, 387-92.

R. Ramanathan, J. A. Zimmerman, and J. R. Eyler, "Ionization Potentials of Small Carbon Clusters," *J. Chem. Phys.* **1993**, *98*, 7838-45.

D. M. Peiris, M. A. Cheeseman, R. Ramanathan, and J. R. Eyler, "Infrared Multiple Photon Dissociation Spectra of Gaseous Ions," *J. Phys. Chem.* **1993**, *97*, 7839-43.

C. H. Watson, J. Wronka, F. H. Laukien, C. M. Barshick, and J. R. Eyler, "The Application of Ultrahigh Mass Resolution to Glow Discharge Mass Spectrometry," *Spectrochimica Acta* **1993**, *48B*, 1445.

*C. H. Watson, J. Wronka, F. H. Laukien, C. M. Barshick, and J. R. Eyler, "High Mass Resolution Glow Discharge Mass Spectrometry using an External Ion Source FT-ICR Mass Spectrometer," *Anal. Chem.* **1993**, *65*, 2801-4.

Chapters Published

S. B. H. Bach, J. E. Bruce, R. Ramanathan, C. H. Watson, J. A. Zimmerman, and J. R. Eyler, "Ionization Potentials and Electron Affinities of Semiconductor Clusters Determined via Charge Transfer Reactions," Book Chapter in Reynolds, P. J., ed., *On Clusters and Clustering: From Atoms to Fractals* Elsevier, Amsterdam, 1993, 59-68.

*S. B. H. Bach, J. E. Bruce, M. A. Cheeseman, R. Ramanathan, C. H. Watson, J. A. Zimmerman, and J. R. Eyler, "Ion/Molecule Reactions of Metal and Semiconductor Clusters: Ionization Potentials and Electron Affinities," Book Chapter in Duncan, M. A., ed., *Advances in Metal and Semiconductor Clusters, Vol II: Cluster Reactions* JAI Press Inc., Greenwich CT, 1994, 41-56.

Most Significant Publications (marked with an * above)

The first of these, "Magic Number" Carbon Clusters: Ionization Potentials and Selective Reactivity," represented the first accurate determination of the ionization potential of C_{60} . Our work, done on laser-generated ions in the gas phase, was later verified by photoelectron spectroscopy on bulk quantities of C_{60} after the facile synthesis of this important compound was reported by Huffman and Kratchmer.

The second publication, "High Mass Resolution Glow Discharge Mass Spectrometry using an External Ion Source FT-ICR Mass Spectrometer," was our demonstration of the highest mass resolution ever attained with a glow discharge source,

better by a factor of 20-50 than the existing commercial instruments.

The third publication, "Ion/Molecule Reactions of Metal and Semiconductor Clusters: Ionization Potentials and Electron Affinities," a chapter in the book *Advances in Metal and Semiconductor Clusters, Vol II : Cluster Reactions* (JAI Press Inc., Greenwich CT, 1994) summarizes almost all the significant results we have obtained on cluster species with ONR support.

Patents

None

Invited Presentations

"Gas Phase Charge Transfer Reactions of Organometallic and Cluster Ions," and "Infrared Multiple Photon Dissociation Studies of Gaseous Ions Using One and Two Lasers," invited seminars presented at the Institute of Chemistry, University of Sao Paulo, Brazil, July, 1992.

"Glow Discharge - FTICR Mass Spectrometry: High Resolution Elemental Analysis," presented by JRE at the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) Annual Meeting, Philadelphia, PA, September, 1992.

"Infrared Multiple Photon Dissociation (IRMPD) in Ion Traps: Determining Infrared Spectra of Gaseous Ions," presented by JRE at the 9th Asilomar Conference on Mass Spectrometry, Pacific Grove, CA, September-October, 1992.

"Laser Formation and Analysis of Gaseous Ions," invited seminar at Chemistry Dept., Auburn University, Auburn AL, February 1993.

"Glow Discharge - FTICR Mass Spectrometry: High Resolution Elemental Analysis," presented by JRE at the Annual Meeting of the Florida Sections of the American Chemical Society, Orlando, FL, May 1993.

"From Metal Atoms to Large Metal Clusters. Current Progress and Future Improvements at High Magnetic Fields," presented by JRE at the 25th Southeast Magnetic Resonance Conference, Gainesville, FL October 1993.

**"Ionization Potentials and Electron Affinities of Carbon Clusters," presented at the Workshop on Gas Phase Ion Chemistry and Physics of Carbon Clusters, Berlin, Germany, March 1994.

"Ultrahigh Resolution Elemental Mass Spectrometry," invited seminar presented to the Chemistry Department, West Virginia University, Morgantown, WV, March 1994.

"Photodetachment and Photodissociation of Gaseous Ions," invited seminar presented to the Analytical Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, TN, April 1994.

"Ultrahigh Resolution Elemental Mass Spectrometry," invited seminar presented to the East Tennessee Mass Spectrometry Discussion Group, Knoxville, TN, April 1994.

"Photodetachment and Photodissociation of Gaseous Ions," invited seminar presented to the Physical Chemistry Division, University of Georgia, Athens, GA, April 1994.

Submitted Presentations

"Electron Affinities and Ion/Molecule Reactions of Carbon and Silicon Clusters," presented by JRE and "Resonance Enhanced Two-Laser Infrared Multiple Photon Dissociation of Gaseous Ions," "Ionization Potentials of Small Carbon Clusters (C_n , $n=3-6$)," "An Improved Ion Guide for External Injection Glow Discharge - Fourier Transform Ion Cyclotron Resonance (GD-FTICR) Mass Spectrometry," and "Glow Discharge Ionization on a High Mass Resolution External Ion Source Fourier Transform Ion Cyclotron Resonance Mass Spectrometer" presented by students and post-docs at the annual ASMS Conference, Washington, DC, June 1992.

"Infrared Multiple Photon Dissociation Spectra of Solvated Ions," and "A Direct Injection Electrospray Source for Fourier Transform Ion Cyclotron Resonance Mass Spectrometry" presented by students at the Annual Meeting of the Florida Sections of the American Chemical Society, Orlando, FL, May 1993.

"Using Ion-Molecule Reactions to Enhance High Resolution GD/FTICR Mass Spectrometry," presented by JRE and "Infrared Multiphoton Dissociation Studies in the Quadrupole Ion Trap using a Multi-Pass Optical Arrangement," "The Application of Glow Discharge-Fourier Transform Ion Cyclotron Resonance (GD-FTICR) Mass Spectrometry to Isotope Ratio Measurements," "Photodissociation of Solvated Ions," "A Direct Injection Electrospray Source for Fourier Transform Ion Cyclotron Resonance Mass Spectrometry," "Infrared Multiple Photon Dissociation Spectra of Solvated Ions," "Hydrogenolysis Rates of Group 4 Methyl Metallocenes," and "Development of a Pulsed-Gas Discharge Ion Source for High Mass Resolution on a Fourier Transform Ion Cyclotron Resonance Mass Spectrometer" presented by students, post-docs, and collaborators at the annual ASMS Conference, San Francisco, CA, June 1993.

Most Significant Presentation (marked with an * above)

The presentation at the Workshop on Gas Phase Ion Chemistry and Physics of Carbon Clusters in Berlin in March, 1994 allowed workers from around the world to gain familiarity with our results on carbon cluster IP's and EA's. In addition, some of our earlier studies were quoted in a number of other presentations. Many requests for reprints/preprints resulted from this presentation.

Technical Reports Published

J. A. Zimmerman, J. R. Eyler, S. B. H. Bach, and S. W. McElvany, ""Magic Number" Carbon Clusters: Ionization Potentials and Selective Reactivity," issued May 10, 1991.

D. K. Bohme, S. Wodek, J. A. Zimmerman, and J. R. Eyler, "Formation of $C_{10}H_8^+$ from the Benzene Radical Cation: A Case for the Growth of Polycyclic Aromatic Hydrocarbon Ions by Ion-Molecule Reactions in the Gas Phase?," issued May 10, 1991.

C. M. Barshick and J. R. Eyler, "A Glow Discharge Ion Source with Fourier Transform Ion Cyclotron Resonance Mass Spectrometric Detection," issued May 10, 1991.

M. A. Cheeseman and J. R. Eyler, "Ionization Potentials and Reactivity of Coinage Metal Clusters," issued May 10, 1991.

J. E. Bruce and J. R. Eyler, "Probing Trapped Ion Energies via Ion-Molecule Reaction Kinetics: Fourier Transform Ion Cyclotron Resonance Mass Spectrometry," issued May 28, 1992.

C. M. Barshick and J. R. Eyler, "An Improved Ion Guide for External Ion Injection in Glow Discharge - Fourier Transform Ion Cyclotron Resonance (GD-FTICR) Mass Spectrometry," issued May 28, 1992.

S. B. H. Bach, J. E. Bruce, R. Ramanathan, C. H. Watson, J. A. Zimmerman, and J. R. Eyler, "Ionization Potentials and Electron Affinities of Semiconductor Clusters Determined via Charge Transfer Reactions," issued May 28, 1992.

S. B. H. Bach, J. E. Bruce, M. A. Cheeseman, R. Ramanathan, C. H. Watson, J. A. Zimmerman, and J. R. Eyler, "Ion/Molecule Reactions of Metal and Semiconductor Clusters: Ionization Potentials and Electron Affinities," issued May 28, 1992.

R. Ramanathan, J. A. Zimmerman, and J. R. Eyler, "Ionization Potentials of Small Carbon Clusters," issued May 5, 1993.

D. M. Peiris, M. A. Cheeseman, R. Ramanathan, and J. R. Eyler, "Infrared Multiple Photon Dissociation Spectra of Gaseous Ions," issued May 5, 1993.

C. H. Watson, J. Wronka, F. H. Laukien, C. M. Barshick, and J. R. Eyler, "Ultrahigh Mass Resolution Glow Discharge Mass Spectrometry: Direct Analysis of Heavy Isotope Mixtures," issued May 5, 1993.

C. H. Watson, J. Wronka, F. H. Laukien, C. M. Barshick, and J. R. Eyler, "High Mass Resolution Glow Discharge Mass Spectrometry using an External Ion Source FT-ICR Mass Spectrometer," issued May 5, 1993.

R. Ramanathan, D. E. Richardson, J. R. Eyler, C. Stacey, G. H. Kruppa, J. Wronka, W. E. Hill, and E. J. Sekabunga, "Electrospray Ionization Fourier Transform Ion Cyclotron Resonance Mass Spectrometric Study of Singly and Doubly Charged Organometallic Complexes," May 1994.

K. L. Goodner, C. Dejsupa, C. M. Barshick, and J. R. Eyler, "High Magnetic Field Glow Discharge Ionization Source," May 1994.

Personnel Funded Under this Grant

Postdoctoral Associates

Dr. George Alameddin (part-time)
Dr. Christopher Barshick (part-time)
Dr. Mitchell Cheeseman (part-time)

Graduate Students

Mr. James Bruce
Mr. Kevin Goodner (summer)
Mr. Tom Hayes (summer)
Mr. Eric Milgram (summer)
Ms. Dil Peiris
Mr. Ragulan Ramanathan
Ms. Yar-Jing Yang

Undergraduates

None

Transitions Resulting from Research under this Grant

Fourier transform ion cyclotron resonance (FTICR) mass spectrometry has demonstrated the highest mass resolving power of any type of mass spectrometry. This is because mass measurement in this technique is directly related to frequency measurement, and frequency is a physical quantity which can be measured with extreme accuracy and precision. Very high mass resolving power is essential in many studies of ions generated by glow discharge (GD) and inductively coupled plasma (ICP) sources. Reliable determination of low levels of contaminants or dopants in a number of materials is often hampered by the presence of "interferants" - species (atomic or polyatomic) of the same nominal mass as that of the species of interest. As part of this ONR-funded research, we carried out collaborative experiments with Dr. Christopher M. Barshick of Oak Ridge National Laboratory and Dr. Clifford H. Watson of Bruker Instruments, Inc., in Billerica, Mass. These experiments demonstrated a mass resolving power of over 650,000 for glow discharge - generated ions, a value **25-50 times higher** than that possible with currently available commercial instruments. For example, we demonstrated that $^{198}\text{Hg}^+$, and $^{198}\text{Pt}^+$, both nominally of mass 198, produced in a glow discharge source from a pressed pin of 10% PtO, 10% HgO and 80% Ag, can easily be resolved from each other. Potential applications of this approach in the analysis of semiconductor, alloy, and meteorite samples are numerous. The mass resolution demonstrated with this approach is more than sufficient for GD-FTICR analysis of the radioactive ^{238}Pu in the presence of much higher levels of ^{238}U , of potential importance in hazardous waste remediation efforts.

Because of a significant potential for future sales, the glow discharge source developed by us and used in the collaborative research described above is **now being offered commercially by Bruker Analytical Systems, Inc.** as an optional ion source for their Fourier transform ion cyclotron resonance mass spectrometers.

FINANCIAL STATUS REPORT (Short Form)

1. Federal Agency and Organizational Element United States Navy		2. Federal Grant or Other Identifying Number Assigned by Federal Agency N00014-87-J-1248		GHS Approval No. 0348-0039	Page of 01 01 Pages
3. Recipient Organization (Name and Complete address, including ZIP code) University of Florida Contracts & Grants Fiscal Services P.O. Box 115650 Gainesville, FL 32611-5650					
4. Employer Identification Number 1596001874C7		5. Recipient Account Number 1606422-12		6. Final Report X Yes No	7. Basis X Cash Accrual
8. Funding / Grant Period (See instructions) From: (Mnth, Day, Year) 07/01/87		To: (Month, Day, Year) 05/31/94		9. Period Covered by this Report From: (Mnth, Day, Year) 10/01/90 To: (Month, Day, Year) 05/31/94	
10. Transactions:		I Previously Reported		II This Report	
a. Total outlays		367,124.22		366,506.83	
b. Recipient share of outlays		0.00		0.00	
c. Federal share of outlays		367,124.22		366,506.83	
d. Total unliquidated obligations		XXX		733,631.05	
e. Recipient share of unliquidated obligations		XXX		0.00	
f. Federal share of unliquidated obligations		XX XXX		0.00	
g. Total Federal share		XXX		733,631.05	
h. Total funds authorized this funding period		XXX		735,229.00	
i. Unobligated balance of Federal funds		XXX		\$1,597.95	
11. Indirect Expense	a. Type of Rate Provisional X Predetermined Final Fixed				
	b. Rate 43% 44% 45%	c. Base \$205,036.35		d.Total Amount \$90,620.05	
12. Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation. ** See Attached					
13. Certification: I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purpose set forth in the award documents.					
Typed or Printed Name and Title Heedley B. Taylor Accounting Coordinator				Telephone (Area code, number and extension) (904) 392-1235	
Signature of Authorized Certifying Official 				Date Report Submitted August 15, 1994	

August 15, 1994

Summary of Overhead:

Agency: Navy N00014-87-J-1248
UF#: 1606422-12

DATES:		DIRECT	LESS	BASE	IDC
10/01/90-06/30/91	45%	89,490.27	45,275.00	44,215.27	19,896.87
07/01/91-06/30/92	43%	51,276.80	8,297.32	42,979.48	18,481.18
07/01/92-06/30/93	44%	86,648.48	7,977.20	78,671.28	34,615.36
07/01/93-05/31/94	45%	48,471.23	9,300.91	39,170.32	17,626.64

DIRECT: \$275,886.78
IDC: 90,620.05
\$366,506.83

**FINANCIAL STATUS REPORT
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a. Total outlays		367,124.22		366,506.83		733,631.05	
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c. Federal share of outlays		367,124.22		366,506.83		733,631.05	
d. Total unliquidated obligations						0.00	
e. Recipient share of unliquidated obligations						0.00	
f. Federal share of unliquidated obligations						0.00	
g. Total Federal share						733,631.05	
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12. Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation. ** See Attached							
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Typed or Printed Name and Title Headley B. Taylor Accounting Coordinator				Telephone (Area code, number and extension) (904) 392-1235			
Signature of Authorized Certifying Official 				Date Report Submitted August 15, 1994			

August 15, 1994

Summary of Overhead:

Agency: Navy N00014-87-J-1248
UF#: 1606422-12

DATES:		DIRECT	LESS	BASE	IDC
10/01/90-06/30/91	45%	89,490.27	45,275.00	44,215.27	19,896.87
07/01/91-06/30/92	43%	51,276.80	8,297.32	42,979.48	18,481.18
07/01/92-06/30/93	44%	86,648.48	7,977.20	78,671.28	34,615.36
07/01/93-05/31/94	45%	48,471.23	9,300.91	39,170.32	17,626.64

DIRECT: \$275,886.78
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